

The Technology and Future of In-Situ Resource Utilization (ISRU)

The University of Central Florida

Oxygen Extraction from Minerals

**Presented by Dr. Tony Muscatello
NASA Applied Chemistry Laboratory
Kennedy Space Center
February 6, 2017**

Outline

- **Introduction**
- **Hydrogen or Carbon Monoxide Reduction of Ilmenite (FeTiO_3)**
- **Carbothermal Reduction of Metal Oxides and Silicates w/Methane**
- **Molten Regolith Electrolysis (MRE)**
- **Combined CO/Carbothermal Reduction of Metal Oxides and Silicates**
- **Water Extraction from Regolith**
- **Recent Development and Field Demonstrations**
- **Challenges**
- **Future Directions**

Introduction

- Lunar regolith contains reducible oxides:

Oxide	Lunar Highland Soils			Lunar Mare Soils	
	JSC-1 Lunar Simulant (wt%)	Lunar Sample 14163 (wt%)	Lunar Sample 62230 (wt%)	Lunar Sample 10002 (wt%)	Lunar Sample 12001 (wt%)
SiO ₂	42.55	47.3	46.6	42.2	46.0
TiO ₂	7.71	1.6	-	7.75	2.8
Al ₂ O ₃	13.47	17.8	26.5	13.6	12.5
Fe ₂ O ₃	3.44	-	-	-	-
FeO	15.16	10.5	5.60	15.3	17.2
MgO	7.98	9.6	6.40	7.76	10.4
CaO	11.99	11.4	14.5	11.9	10.9
Na ₂ O	0.45	0.7	0.44	0.47	0.48
K ₂ O	0.15	0.6	-	0.16	0.26
MnO	0.21	0.1	-	0.20	0.22
Cr ₂ O ₃	-	0.2	-	0.30	0.41
P ₂ O ₅	0.14	-	-	0.05	-

- But they are very stable and require lots of work!

Propellant from the Moon will revolutionize our current space transportation approach

Each Apollo mission utilized Earth-derived propellants (Saturn V liftoff mass = 2,962 tons)

What if lunar lander was refueled on the Moon's surface?
73% of Apollo mass (2,160 tons)

Assume refueling at L1 and on Moon: 34% of mass (1,004 tons)

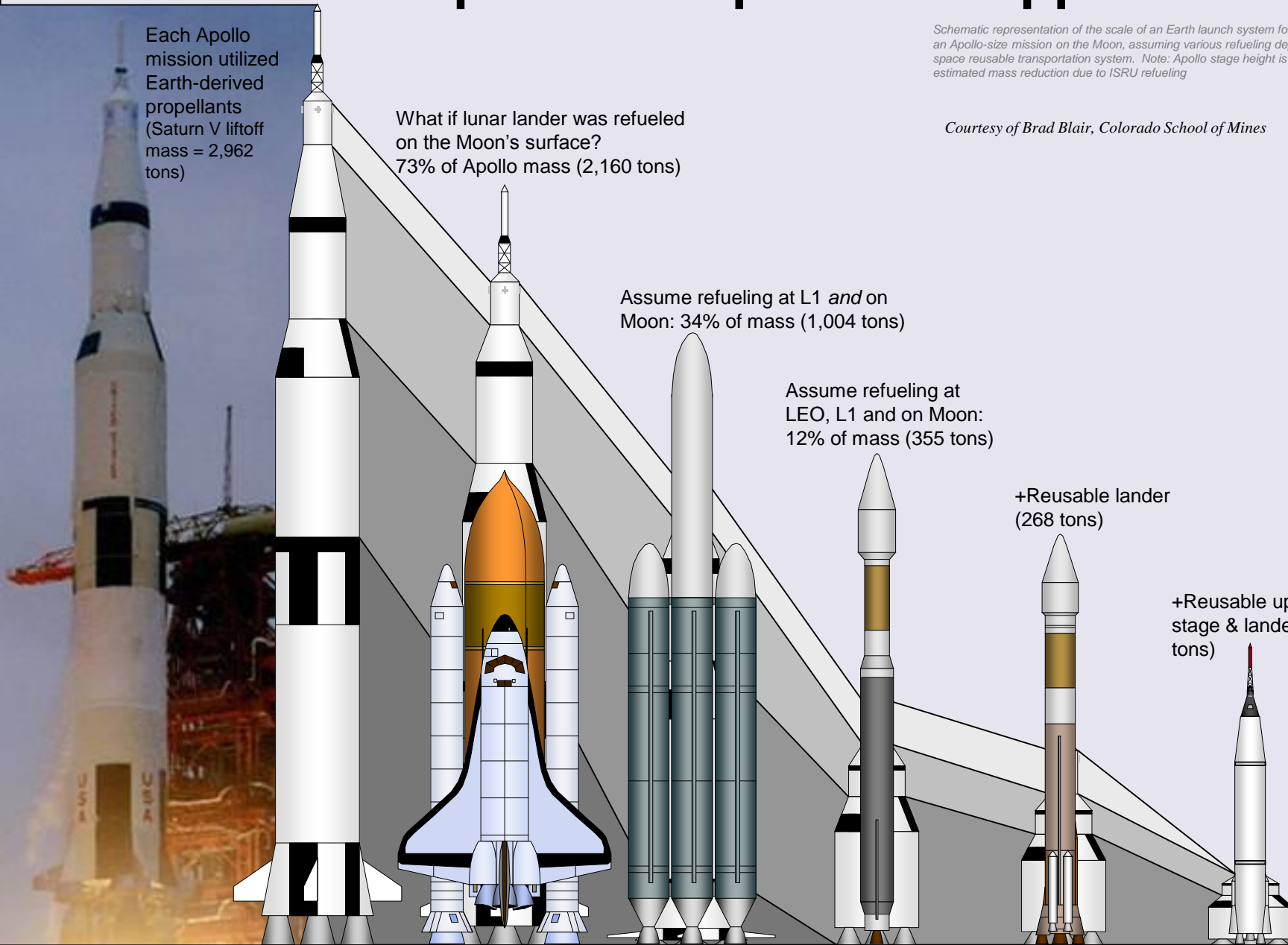
Assume refueling at LEO, L1 and on Moon: 12% of mass (355 tons)

+Reusable lander (268 tons)

+Reusable upper stage & lander (119 tons)

Schematic representation of the scale of an Earth launch system for scenarios to land an Apollo-size mission on the Moon, assuming various refueling depots and an in-space reusable transportation system. Note: Apollo stage height is scaled by estimated mass reduction due to ISRU refueling

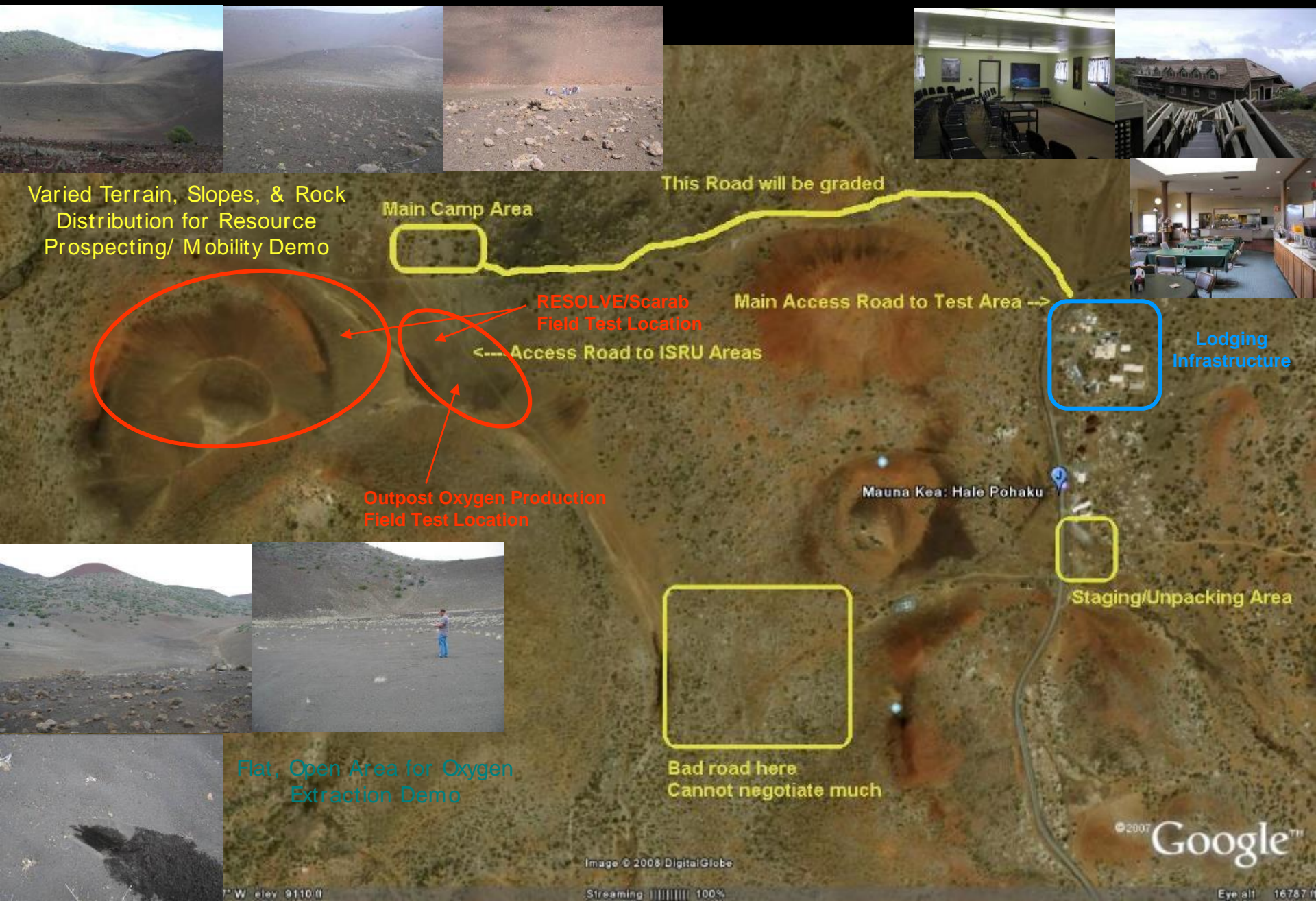
Courtesy of Brad Blair, Colorado School of Mines



Hydrogen or Carbon Monoxide Reduction of Ilmenite (FeTiO_3) and Pyroclastic Glass-Chemistry

- Lunar regolith and regolith at other locations (asteroids and Mars) contain a variety of metal oxides in the form of oxides, silicates, and aluminates
- Lunar regolith contains ilmenite (FeTiO_3) at 0.4-12.8 vol% depending on location, with lunar mare being higher than the highlands
- H_2 Reduction: $\text{H}_2 + \text{FeTiO}_3 \rightarrow \text{H}_2\text{O} + \text{Fe}^0 + \text{TiO}_2$ ($T = 900^\circ\text{C}$)
 $\text{H}_2\text{O} + 2 e^- \rightarrow \text{H}_2 + \frac{1}{2} \text{O}_2$
 O_2 Yield = ~1-2 wt% - depending on location
- CO Reduction: $\text{CO} + \text{FeTiO}_3 \rightarrow \text{CO}_2 + \text{Fe}^0 + \text{TiO}_2$ ($T = 900^\circ\text{C}$)
 $\text{CO}_2 + \text{H}_2 \rightarrow \text{H}_2\text{O} + \text{CO}$ (RWGS Reaction)
 $\text{H}_2\text{O} + 2 e^- \rightarrow \text{H}_2 + \frac{1}{2} \text{O}_2$
 O_2 Yield = ~1-2 wt% - depending on location

2008 ISRU Field Test Infrastructure and Test Layout – Mauna Kea

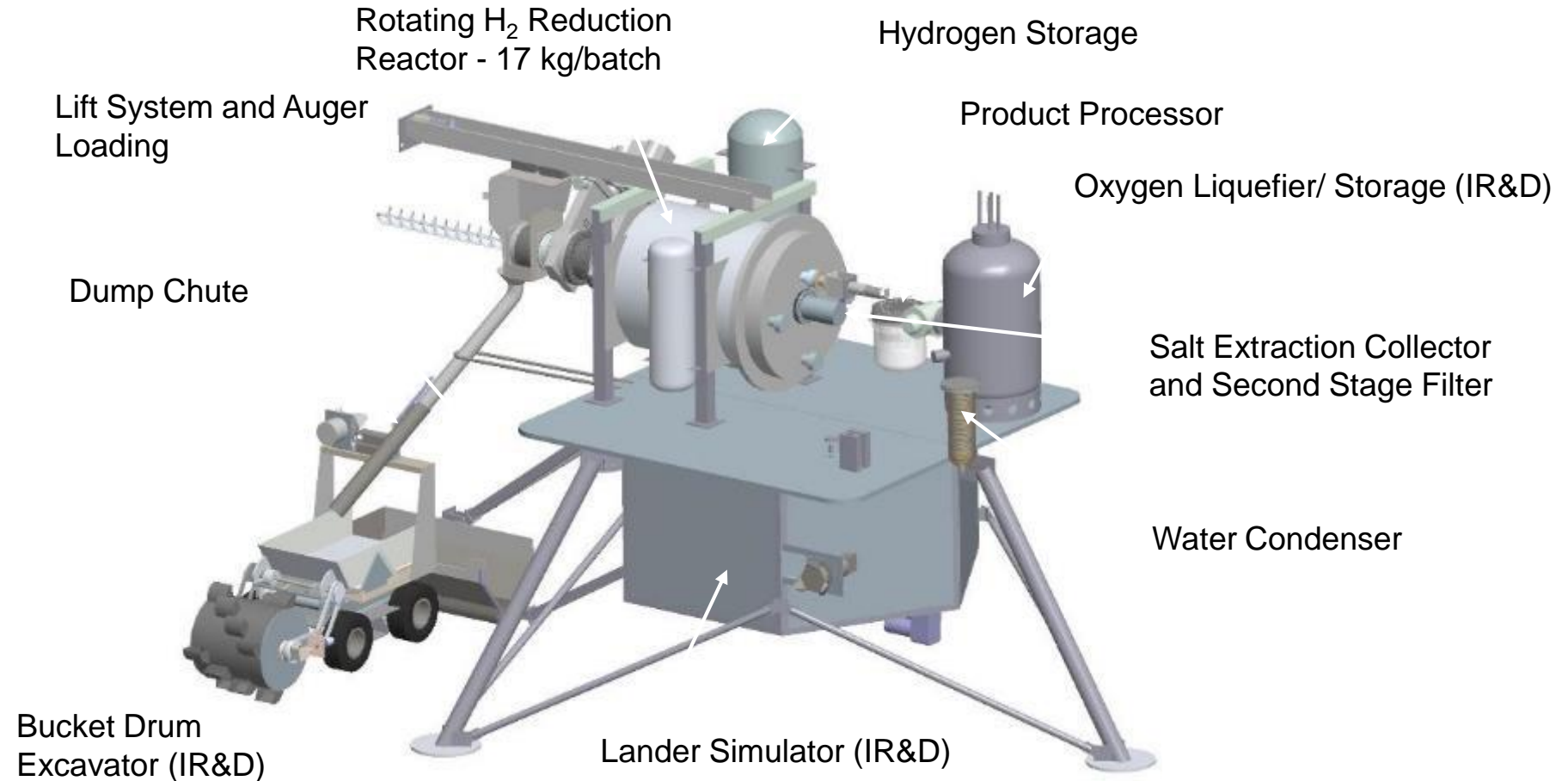






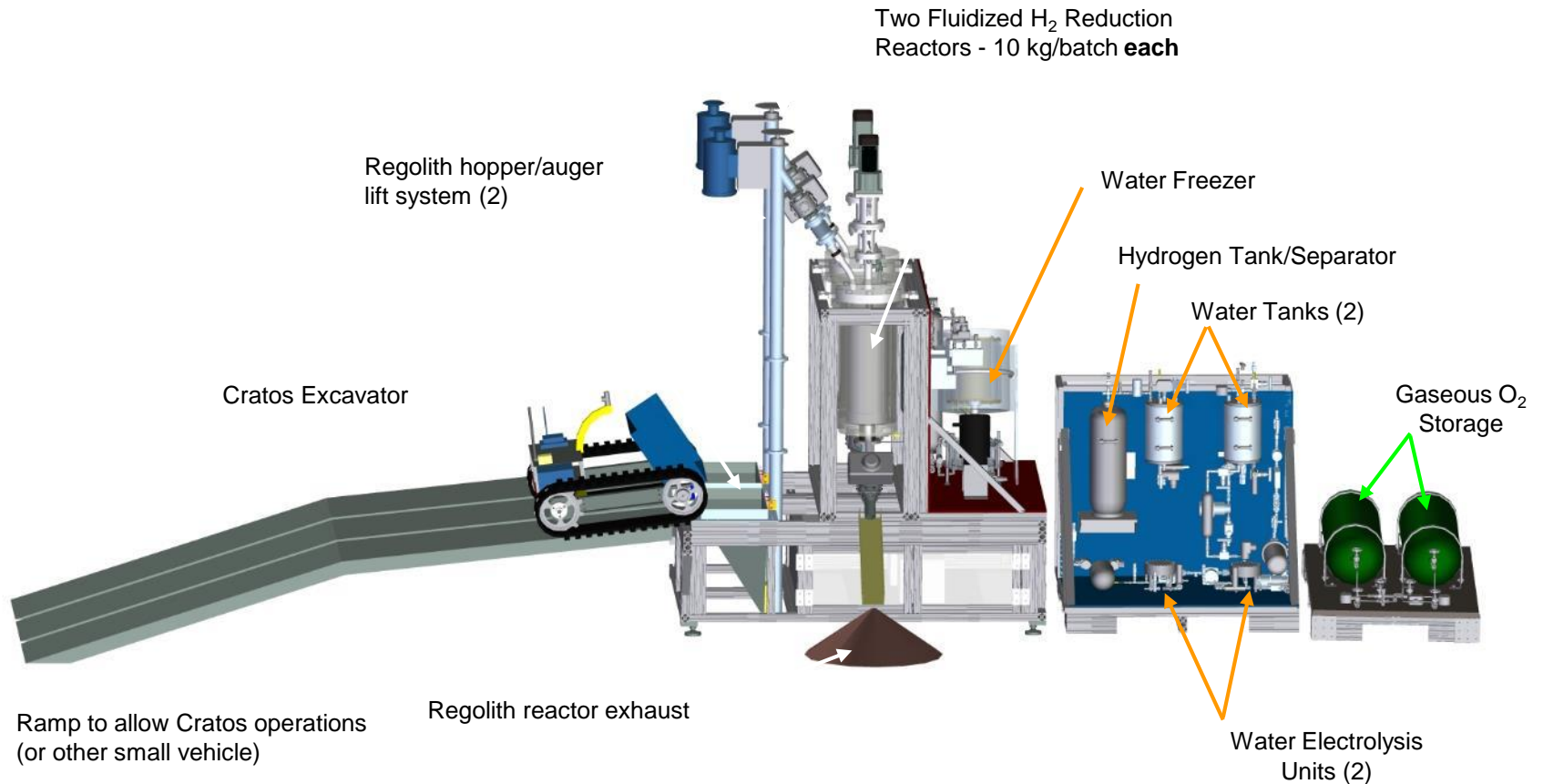


PILOT Field Test Hardware



**PILOT – Precursor ISRU Lunar Oxygen Testbed
(Lockheed Martin Astronautics – Denver)**

ROxygen Field Test Hardware

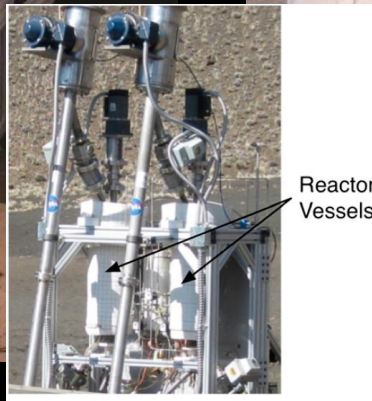
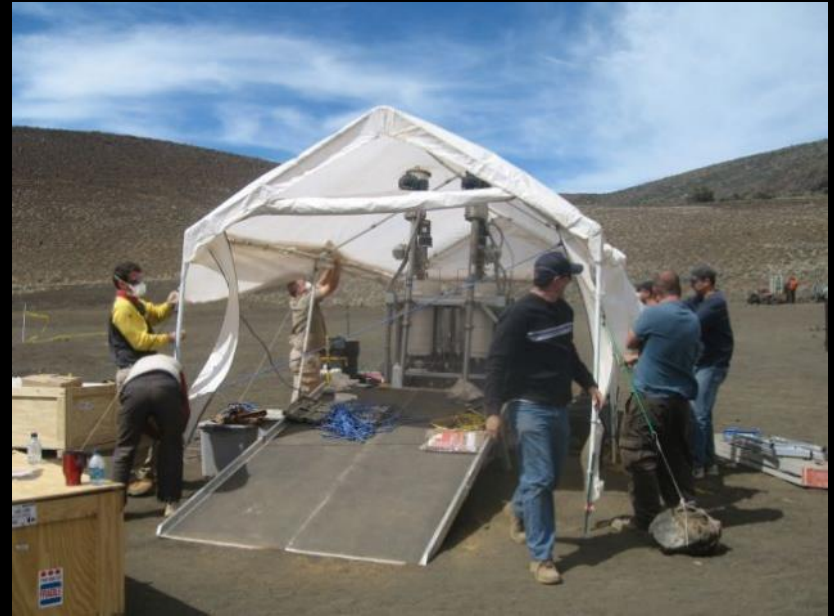
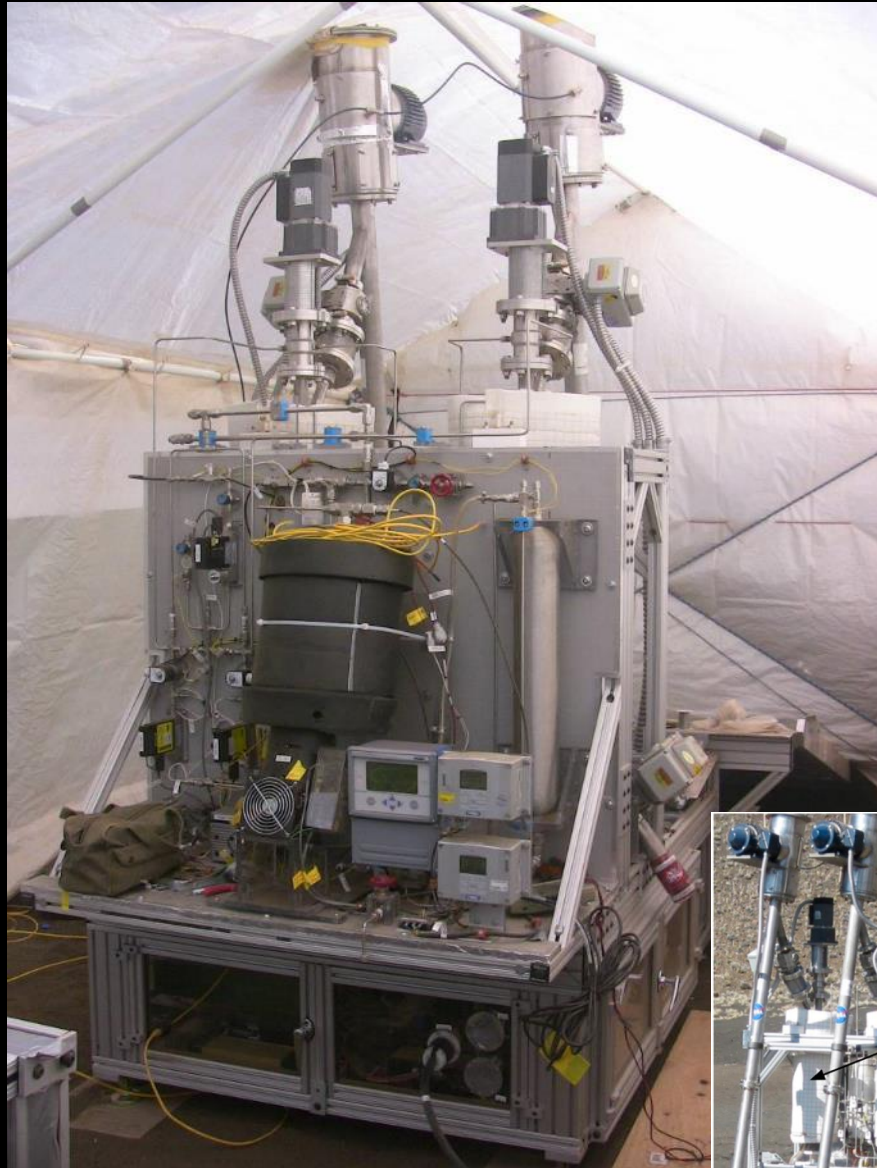


NASA ROxygen H₂ Reduction System
(JSC, KSC, and GRC)

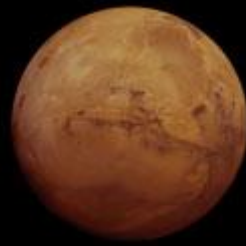
ROxygen & PILOT Tasks

- Demonstrate excavation and material delivery to plant and removal of spent regolith;
 - Increase distance and terrain complexity between plant and excavation site each day
- Demonstrate regolith processing to extract oxygen
 - Min. of 4 h on one day; nominal 8 h per day
 - Max. of 8 h/day for 5 days
- Demonstrate oxygen separation and storage
 - Liquefaction and cryogenic storage
 - Moderate pressure gaseous oxygen
- Opportunistic Demos
 - Demonstrate alternative oxygen liquefaction and storage
 - Hot fire a LO_2/LCH_4 RCS 25 lbf thruster igniter
 - Mossbauer spectrometer on Cratos to measure iron before and after processing

ROxygen Setup



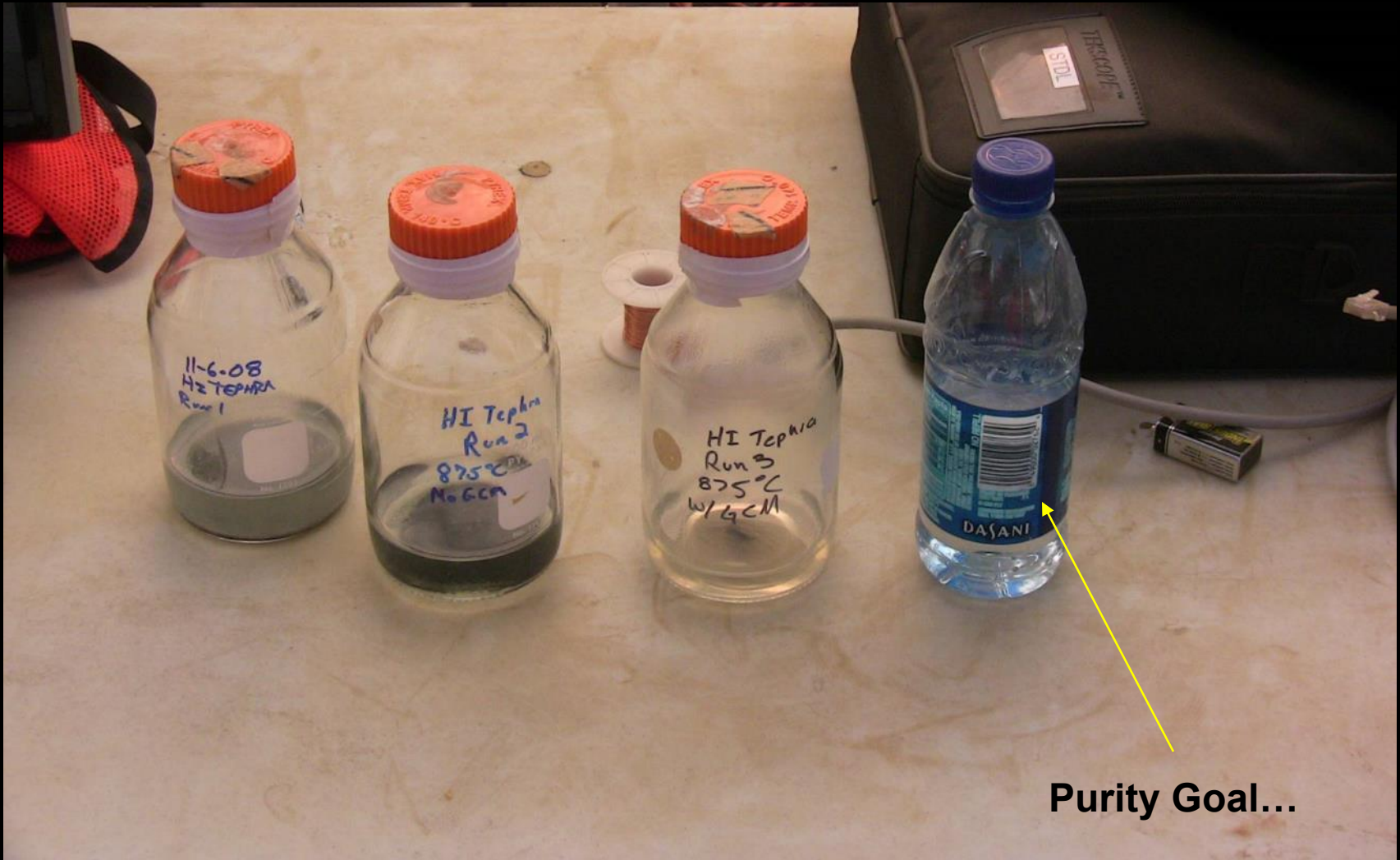
PILOT



2008 Field Test Results

- **PILOT (1000 kg O₂ per year design scale)**
 - System integrated with mini rover equipped with bucket drum excavator bringing soil to the reactor. Integrated system was tested during field tests in Hawaii.
 - Processing capacity of ~15 kg of regolith per batch yielding 150 g of O₂.
 - Produced oxygen yields of 1 wt% of processed regolith.
- **ROxygen (~667 kg O₂ per year design scale)**
 - Two large-scale reactors were tested in 2008 at the first ISRU technology field tests.
 - Tests in large reactor provided oxygen yields in the range 0.2% – 0.5% by mass of regolith. The low yields are attributed to the operational limits of a contaminant scrubber.
 - Recirculation of hydrogen was achieved with removal of hydrogen halide contaminants from the produced water.

Water From Rocks!



Purity Goal...

More info at: <https://isru.nasa.gov/Hydrogen-Reduction-of-Regolith.html>

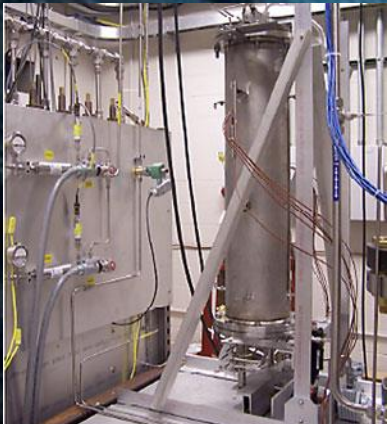
Concentric Hydrogen Reduction Reactor

- **Technology features**

- Concentric cylindrical chambers allow the exchange of heat from the hot regolith being processed to fresh regolith waiting to be introduced.
- Maintains regolith in fluid and loose state (fluidization) by hydrogen flow and/or vibration of the reactor – compares efficiencies.

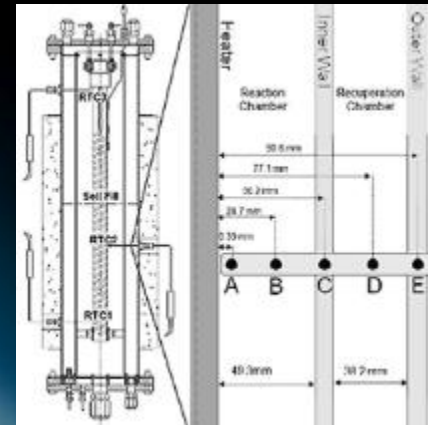
- **Results and Performance**

- Vibrofluidization and gas fluidization with Helium gas are near equally efficient in heat recuperation. Fluidization with hydrogen is not as effective.
- Tests in large reactor provided oxygen yields in the range 0.2% – 0.5% by mass of regolith. The low yields are attributed to the operational limits of a contaminant scrubber.
- Added mass and complexity of a dual chamber reactor is also counterproductive for ISRU usage.



Dual Chamber H₂ reduction reactor mounted on flex stand (Credit: Glenn Research Center)

Hardware schematic for dual chamber H₂ reduction reactor with thermocouple locations (credit: Glenn Research Center).



Carbothermal Reduction of Metal Oxides and Silicates - Chemistry

- Methane Decomposition and Carbon Deposition:



- Carbothermal Reduction:



- Methane Regeneration (Sabatier Rxn):

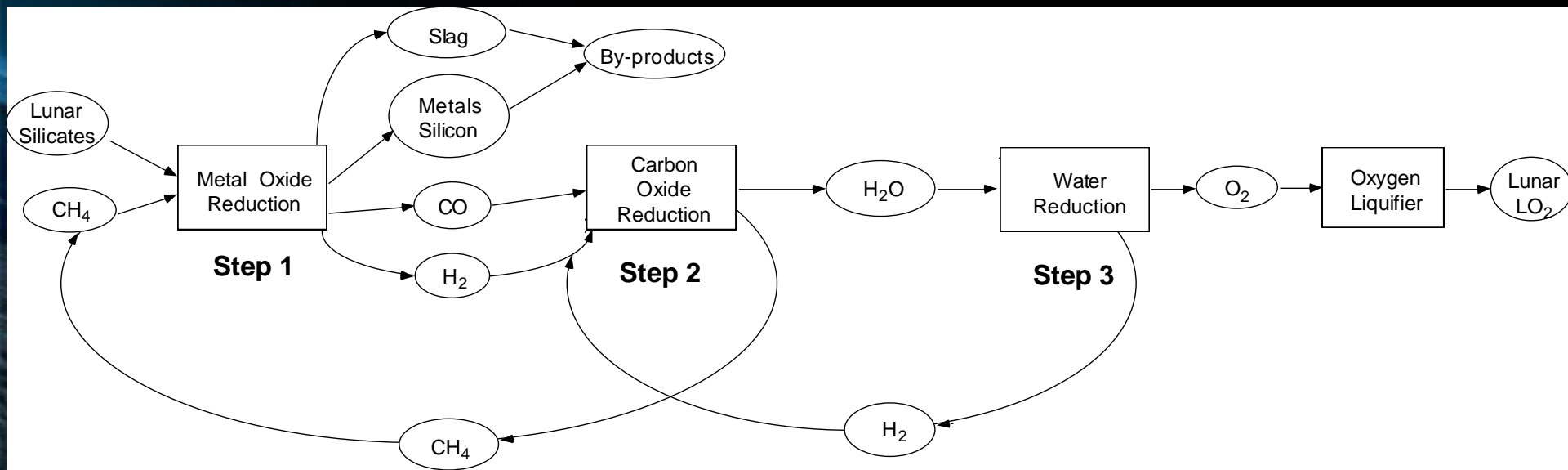


- Water Electrolysis:

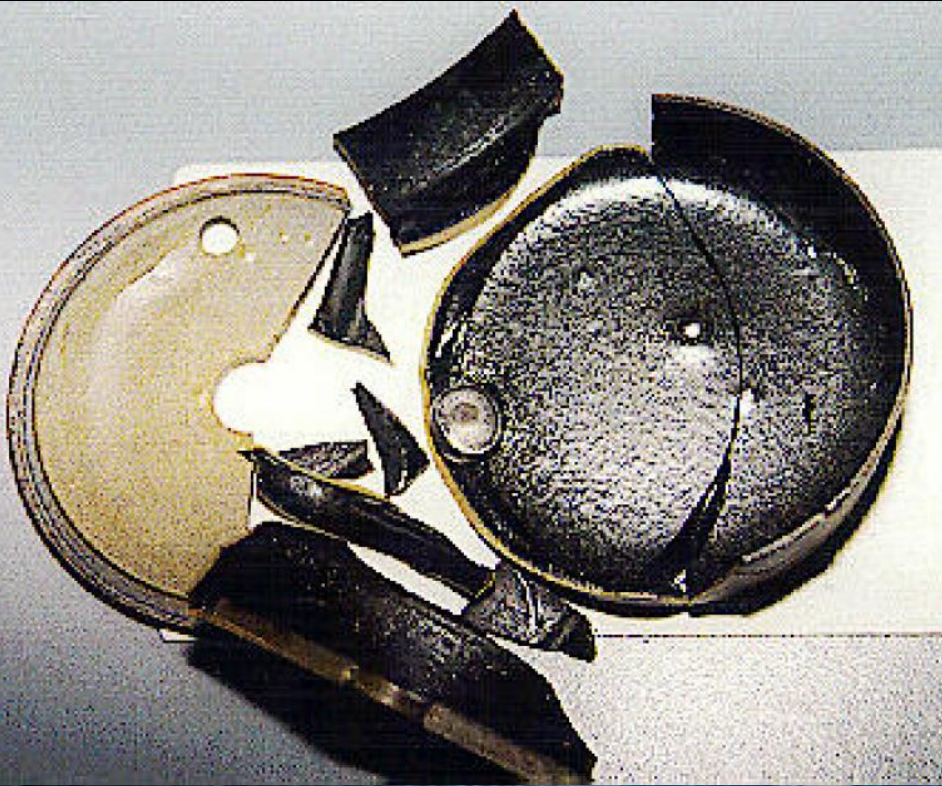


O_2 Yield = <15 wt% with acceptable carbon losses

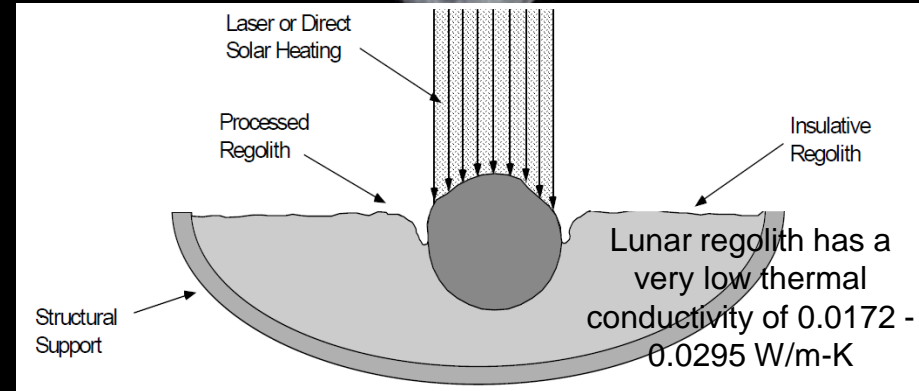
Carbothermal Reduction of Silicates – Reaction Summary



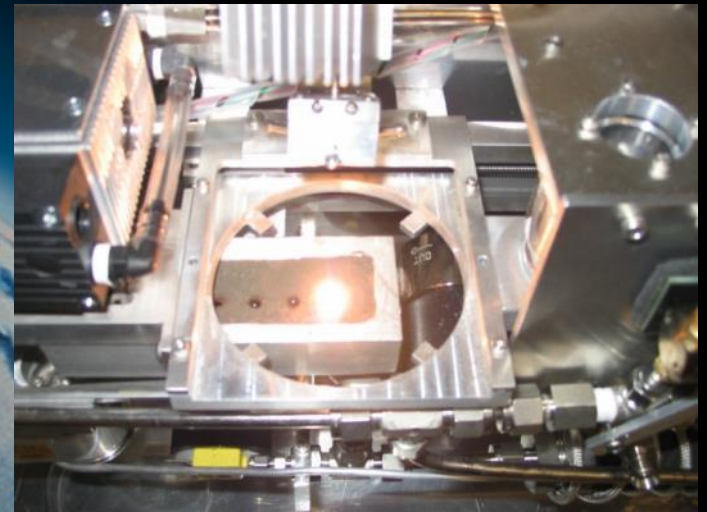
Carbothermal Reduction of Silicates – Practical Limitations and Solution



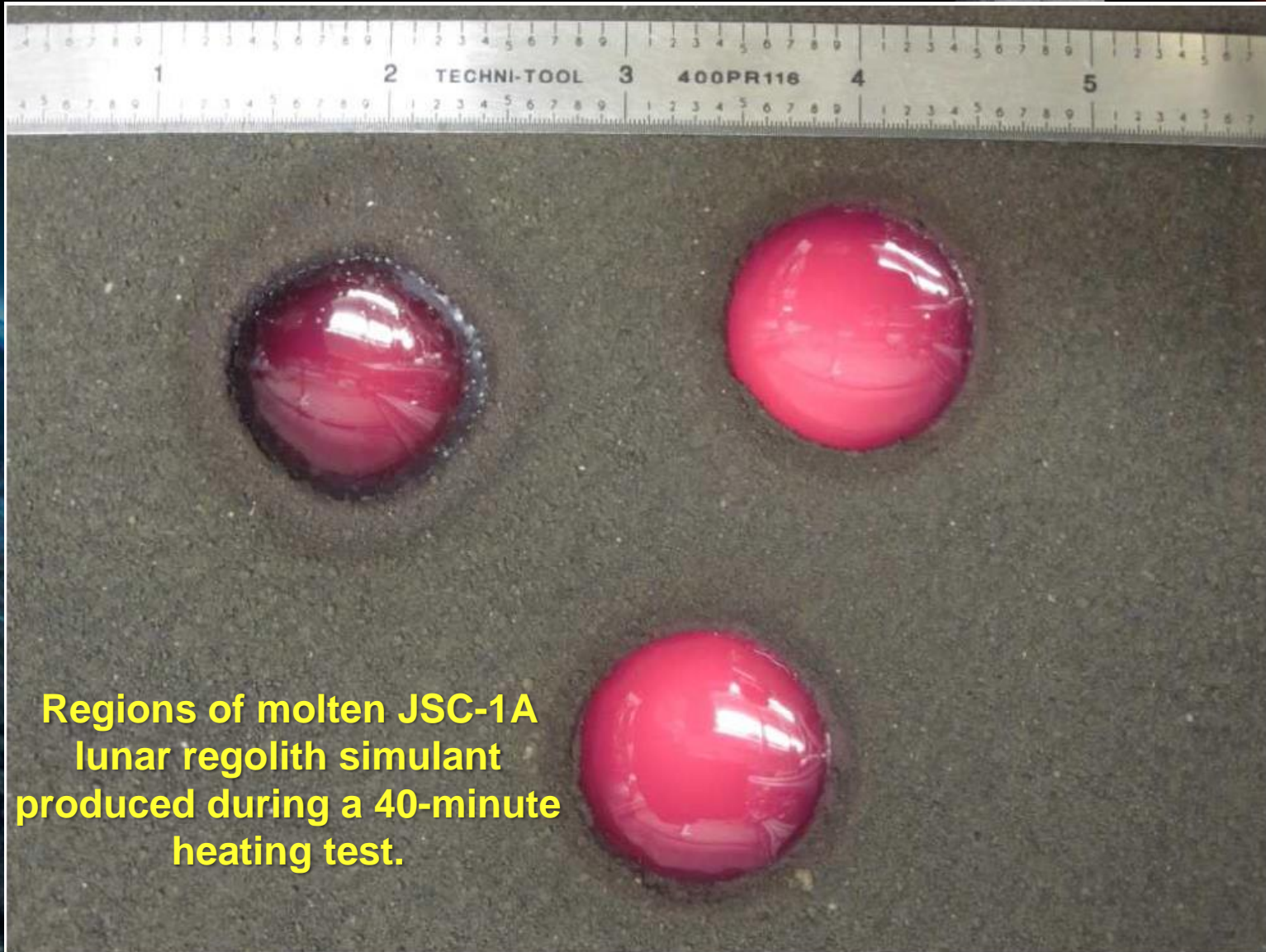
Typical crucible after a single carbothermal reduction processing batch in a traditional hot-wall furnace.



Direct energy processing approach where the regolith becomes its own processing container.

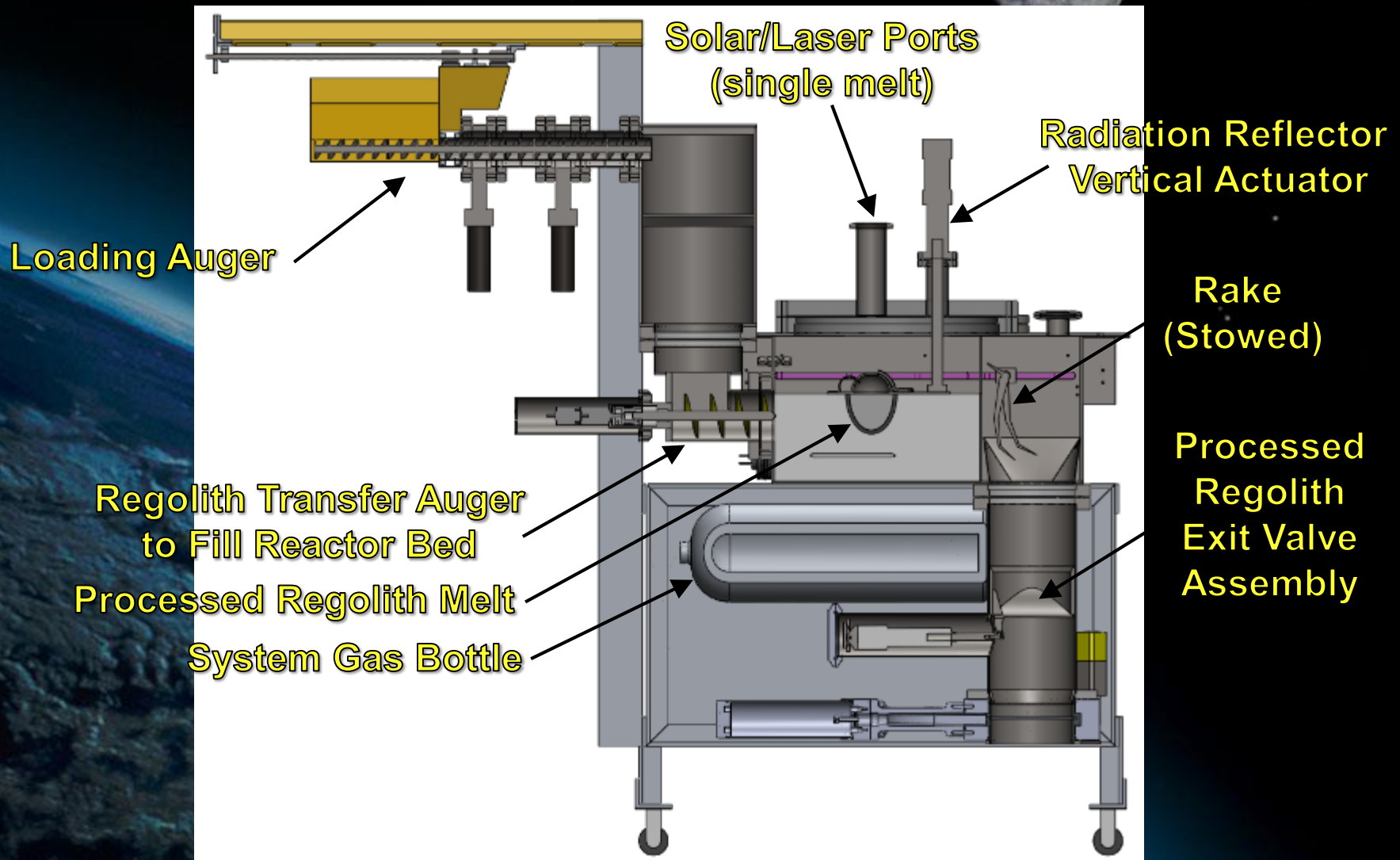


Carbothermal Reduction – 3 x 470 W CO₂ Laser Tests

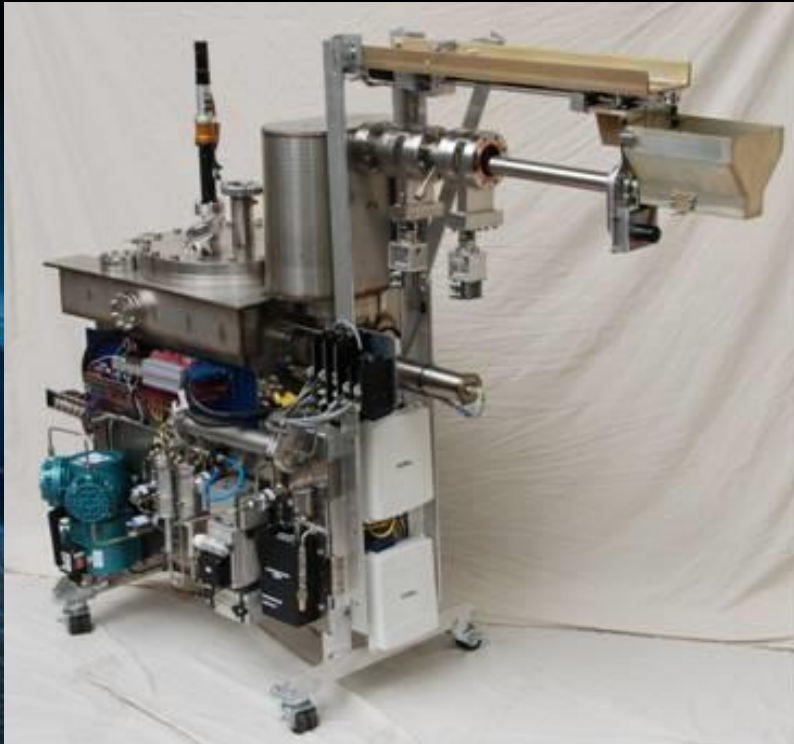


**Regions of molten JSC-1A
lunar regolith simulant
produced during a 40-minute
heating test.**

Carbothermal Regolith Reduction Module (ORBITEC)



Carbothermal Regolith Reduction Module



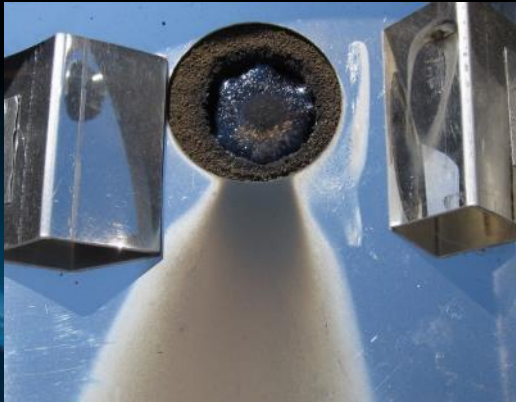
- **ORBITEC developed the Carbothermal Regolith Reduction Module to demonstrate remote, semi-autonomous extraction of oxygen from lunar regolith simulant using solar energy**
 - Automated filling of the regolith hopper and transfer to carbothermal reduction reactor
 - Automated gas handling system, including gas clean-up beds and methanation reactor
 - Automated removal of processed regolith
 - Ability to operate remotely through an <http://> interface
- Hardware is sized to support oxygen production at a rate 1 MT O₂ per year

Solar Energy Collection and Delivery Module

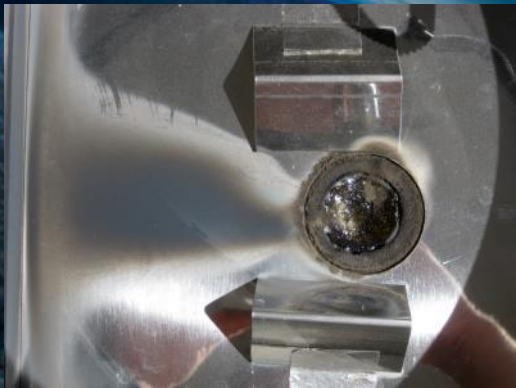


- Physical Sciences Inc. built a Solar Energy Collection and Delivery Module that provides concentrated solar energy to the Carbothermal Regolith Reduction Module
- Seven solar concentrators are mounted on an array with two-axis tracking of the sun
- The solar energy from each concentrator is delivered to the carbothermal reactor via a fiber optic cable
- Each fiber optic cable delivers 86 to 100 W (total of 600 to 700 W) of concentrated solar energy

Carbothermal Reduction with Solar Energy

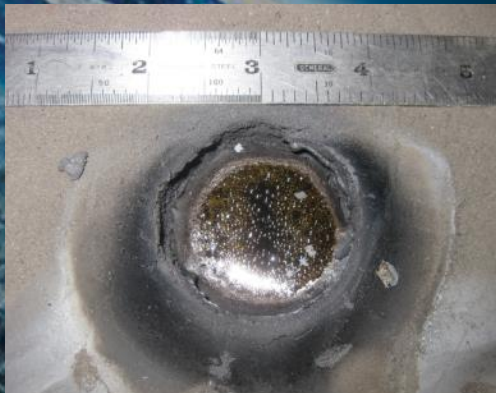


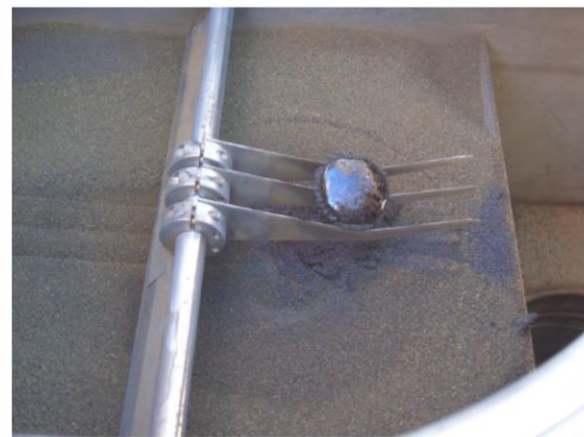
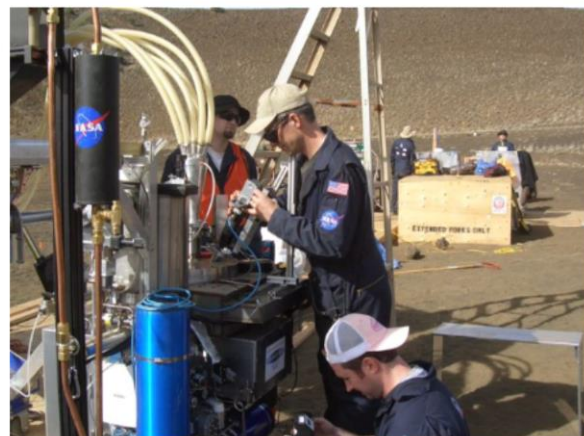
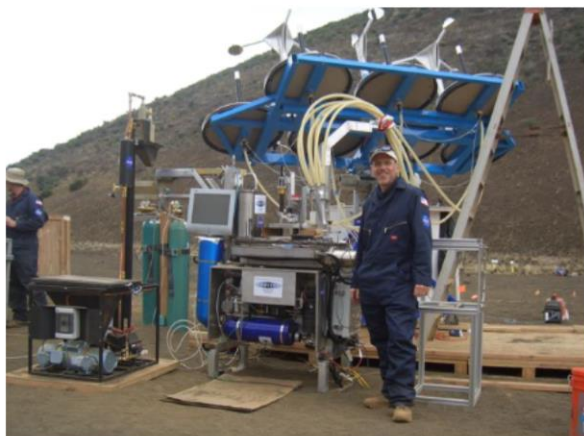
Processed Tephra



*Processed JSC-1A
Simulant*

- Oxygen yields up to 10.3%wt with processing times up to 80 minutes have been demonstrated (JSC-1A lunar regolith simulant and tephra)
- The carbothermal reduction process produces silica fume as an intermediate product, so keeping the end of the quartz rod clean has been a challenge
- ORBITEC has built and successfully tested a gas nozzle that keeps the end of the quartz rod clean during processing (aerodynamic window)





**Solar Concentrator/
Carbothermal Reduction of
Regolith for Lunar Oxygen
Production – 2010 Field
Demo on Mauna Kea
NASA/PSI/ORBITEC**



Carbothermal Reduction with Solar Energy – Field Test Results



Carbothermal Regolith Reduction Module, Solar Energy Module and Pneumatic Regolith Transfer Unit during the 2010 ISRU Analog Test.

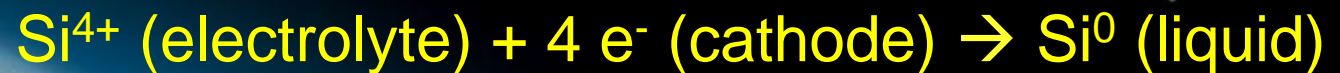
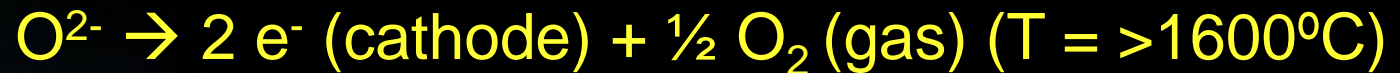
- The carbothermal plant
 - Performed 17 tephra melts (12 for O₂ production)
 - over 7 days
 - Produced 31.5 g of water from the tephra
- Equivalent to 28 g of oxygen
 - 9.7% oxygen yield by mass



Processed tephra melt from Day 10 – Experiment 2

Molten Regolith Electrolysis - Chemistry

- Molten Oxide Electrolysis:

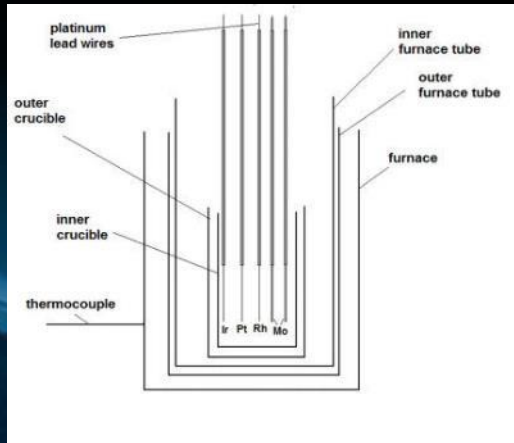


O_2 Yield = 15-37 wt% depending on scale of operation and feed (mare vs. highlands)

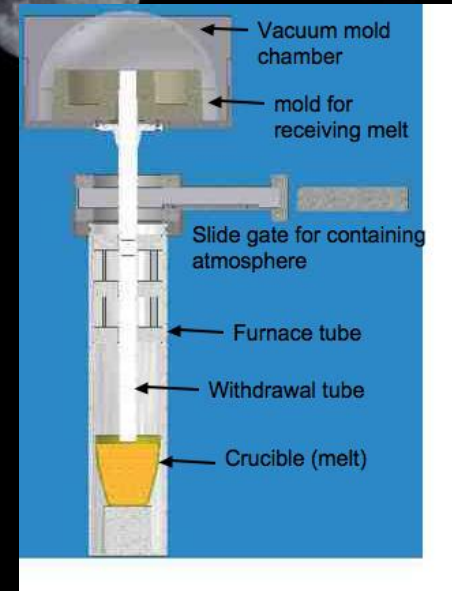
Metals: [Will be covered by Laurent Sibille]

More info at https://isru.nasa.gov/Molten_Regolith_Electrolysis.html

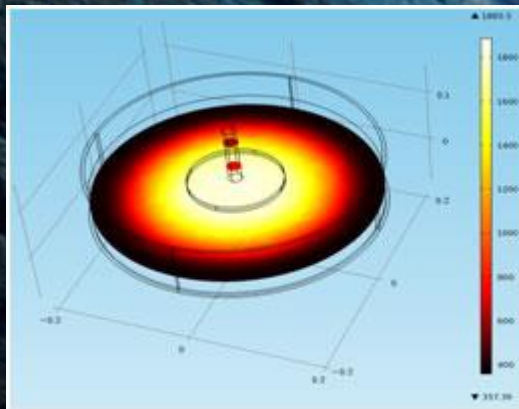
Molten Regolith Electrolysis



Schematic of electrolytic cell configuration to investigate electrochemical behaviors of various anode materials in molten oxides (left) and furnace containing the cell in operation at 1600°C (right) (MIT)



Schematic of counter gravity Molten Material Withdrawal device (Ohio State U.)



Thermal model of Joule heating in a Molten Regolith Electrolysis reactor in which the regolith itself contains a centered pool of electrolyte heated by the electrolytic current. (Image credit: NASA Kennedy Space Center)



Small casting of molten ferrosilicon (lower layer) and molten oxide of lunar composition (top layer) withdrawn by counter-gravity suction at 1600°C from reactor furnace (Image credit: Ohio State U./KSC)

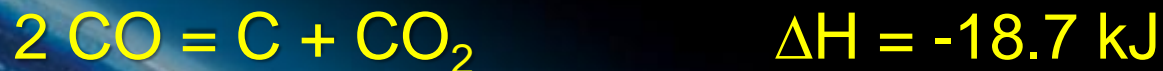
Combined CO/Carbothermal Reduction of Metal Oxides and Silicates – Chemistry (Pioneer Astronautics)

- Carbon Monoxide Silicate Reduction System (COSRS):

Iron oxide reduction ($T = 800\text{-}850^{\circ}\text{C}$):



Carbon deposition (carbon monoxide disproportionation) ($T = 600^{\circ}\text{C}$):



Carbothermal reduction ($T = \text{up to } 1600^{\circ}\text{C}$):



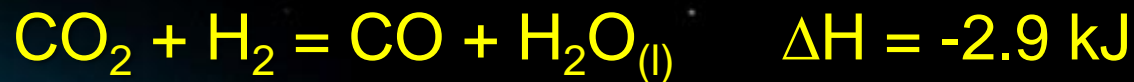
More info at

http://www.lpi.usra.edu/meetings/leag2005/presentations/wed_am/04_berggren.pdf

Combined CO/Carbothermal Reduction of Metal Oxides and Silicates – Chemistry (Cont.)

- Carbon Monoxide Silicate Reduction System (COSRS):

Reverse water gas shift reaction ($T = 400^{\circ}\text{C}$):



Electrolysis:



O_2 Yield = ~15-20 wt%

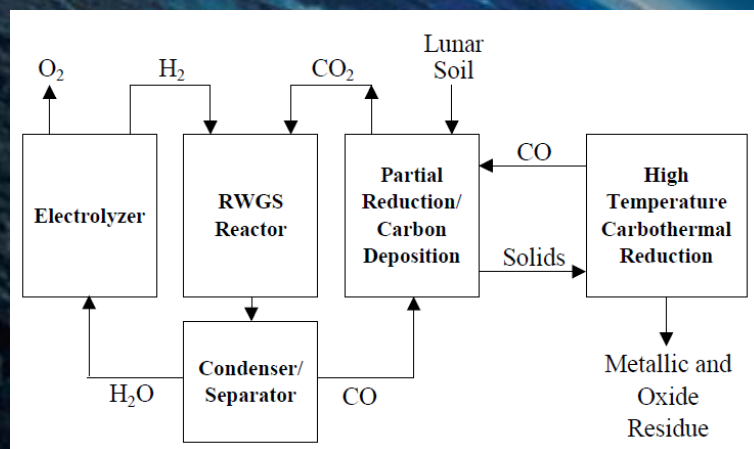
Byproducts: SiO (up to 5 wt%) can be reduced to nearly pure Si metal

Ferrosilicon alloy – up to 25 wt%

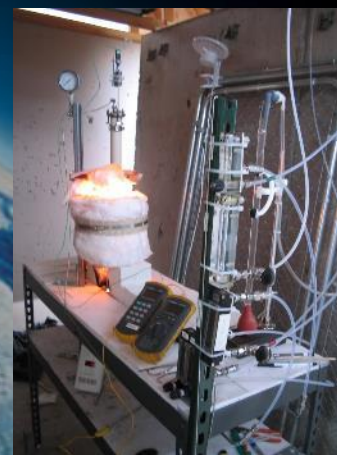
Combined CO/Carbothermal Reduction of Metal Oxides and Silicates

Recoverable Oxygen in JSC-1 Lunar Simulant

Compound	kg Compound per 100 kg Soil	Compound Molecular Weight	kg Oxygen per 100 kg Soil	Cumulative kg Oxygen per 100 kg	kg Carbon per 100 kg Soil	Cumulative kg Carbon per 100 kg
Fe ₂ O ₃	3.44	159.70	1.03	1.03	0.78	0.78
FeO	7.35	71.85	1.64	2.67	1.23	2.00
P ₂ O ₃	0.66	110.00	0.29	2.96	0.22	2.22
Cr ₂ O ₃	0.04	152.00	0.01	2.97	0.01	2.23
MnO	0.18	71.00	0.04	3.01	0.03	2.26
SiO ₂	47.71	60.00	25.45	28.46	19.08	21.34
TiO ₂	1.59	79.88	0.64	29.09	0.48	21.82



COSRS Process Description



Carbothermal Reduction Furnace



Iron Oxide Reduction
- Carbon Deposition
Reactor (81 g JSC-1)

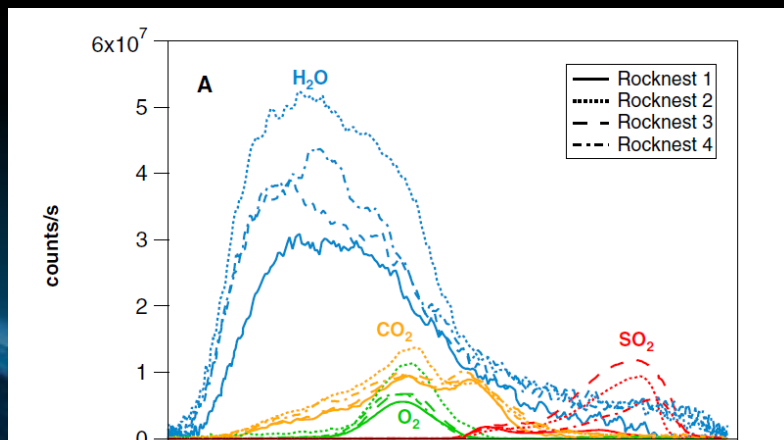
Lunar JSC-1 Carbothermal Residue



Combined CO/Carbothermal Reduction of Metal Oxides and Silicates – (Berggren 2005)

- “Identified conditions leading to maximum leverage (= O₂ recovered/C lost).
 - Carbon losses are mostly as silicon carbide.
 - Carbon losses can be mitigated by reacting silicon carbide with fresh silica in the lunar soil as follows.
$$\text{SiO}_2 + 2 \text{SiC} = 3 \text{Si} + 2\text{CO} \quad \Delta H = 833.6 \text{ kJ}$$
- Similarly, silicon carbide can be reacted with fresh ferrous oxide in the lunar soil as follows.
$$\text{FeO} + \text{SiC} = \text{Si} + \text{Fe} + \text{CO} \quad \Delta H = 228.6 \text{ kJ}$$
- The above reactions suggest that adding less than the theoretical, or stoichiometric, amount of carbon to reduce all of the silicon oxide might also reduce carbide formation.”
- Additional work has not been published yet.

Water Extraction from Regolith

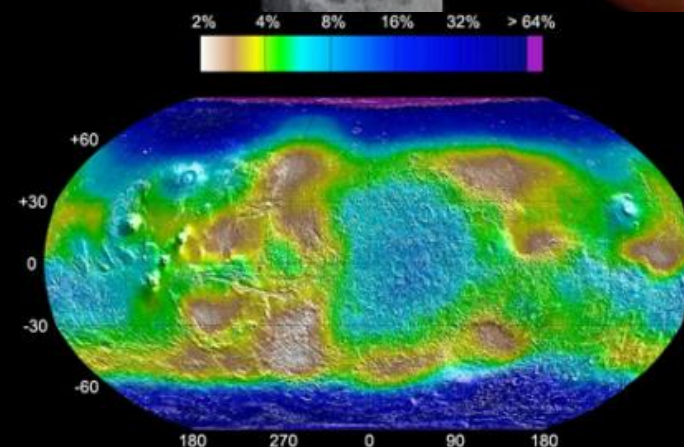


	Run 1	Run 2	Run 3	Run 4
CO ₂	8.3 ± 2.0	10.8 ± 2.6	10.1 ± 2.4	10.4 ± 2.5
SO ₂	2.9 ± 0.2	13.7 ± 1.9	21.7 ± 2.9	10.5 ± 1.4
H ₂ O	43.3 ± 10.7	66.5 ± 16.2	54.5 ± 9.9	55.9 ± 11.9
O ₂	3.0 ± 0.4	5.1 ± 0.6	3.7 ± 0.4	3.7 ± 0.5

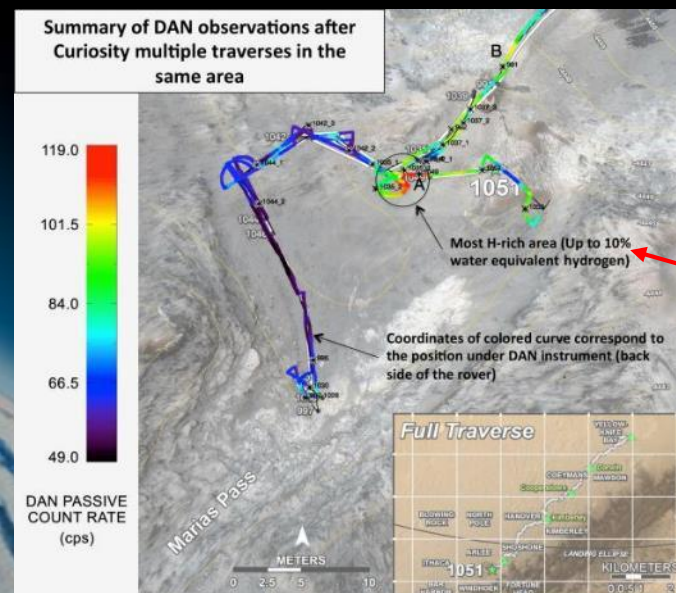
	Sample weight %			
	Run 1	Run 2	Run 3	Run 4
CO ₂	0.7 ± 0.2	1.0 ± 0.3	0.9 ± 0.3	0.9 ± 0.3
SO ₃ equiv.	0.5 ± 0.1	2.2 ± 0.5	3.5 ± 0.7	1.7 ± 0.3
H ₂ O	1.6 ± 0.5	2.4 ± 0.7	2.0 ± 0.5	2.0 ± 0.5
ClO ₄ equiv.	0.3 ± 0.1	0.5 ± 0.1	0.4 ± 0.1	0.4 ± 0.1

Gases released from heated Rocknest aliquots

Volatile, Isotope, and Organic Analysis of Martian Fines with the Mars Curiosity Rover
 L. A. Leshin *et al.*
Science **341**, (2013);
 DOI: 10.1126/science.1238937



Water in top 1-m on Mars



Up to
10%
Water

Hydrogen-rich area detected at "Marias Pass" on Mars by Curiosity

Challenges

- H₂ Reduction
 - Removal of H₂S and HF from water
 - Scale-up
 - Reactor seals
- Carbothermal Reduction
 - Carbon losses
 - Mass of solar concentrator and dust accumulation
 - Silica fume control
- Molten Regolith Electrolysis
 - Electrode consumption
- COSRS
 - Scale-up and development
- Funding
 - Moon vs. Mars vs. Asteroids
 - All of the above?



Future Direction [Tony's Wish List]

- NASA has restarted its ISRU Project
- NASA should fully embrace ISRU
- Integrated human exploration of the inner solar system
- Moon, asteroids, Mars moons, Mars
- Commercial development of key technologies in partnership w/NASA to reduce costs
 - i.e. shared funding
 - Habitats, ISRU, spacecraft, launch vehicles, etc.
 - Asteroid mining
- Pick an architecture and stick with it for >8 years